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METHOD FOR WASHING CHANNEL OF AUTO-SAMPLER AND AUTO-SAMPLER
[Autosampura no ryuurosenjouhouhou oyobi autosampura]

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(54) [Title of the Invention]

METHOD FOR WASHING CHANNEL OF AUTO-SAMPLER AND AUTO-SAMPLER

(57) [Claims]

[Claim 1] A method for washing the channel of an auto-sampler wherein a needle which has absorbed the sample solution established at the tip of the trial liquid absorption tube is inserted into the washing liquid, absorbing out this washing liquid by approximately the same operation as absorbing in the trial liquid to the sample liquid absorbing in tube by means of the needle, and

next, inserting the needle into a washing port providing a discharge outlet, and under these conditions emitting the washing liquid by making flow the washing liquid in a reverse direction to the absorbing direction to the washing port outside of the needle, and

the washing liquid which was emitted, by absorption processing, is discarded forcibly from the discharge outlet.

[Claim 2] A method for washing the channel of an auto-sampler according to Claim 1 wherein a plurality of different washing liquids are prepared, and along with sequential insertion of the needle within this plurality of washing liquids the washing liquid is absorbed in and emitted out.

[Claim 3] In an auto-sampler having a sample liquid absorbing tube on which a needle is mounted at the tip, a sample bottle which

* Paragraph numbers take place for the original pagination in the foreign text.

stores the sample liquid for absorption through the needle, and a path which supplies to a column the sample liquid which is absorbed by the needle comprising a washing bottle for storing the washing liquid, a washing port which provides a discharge outlet, and an absorption means connected to the discharge outlet, and after temporarily absorbing the washing liquid stored in the washing bottle by means of the needle which has absorbed the sample liquid, possibly discharging the absorbed washing liquid, together with moving to within the washing port, within the washing port from the needle, and in addition, absorbing the washing liquid emitted to the washing port from the needle by means of the absorbing means, possibly strongly discharging.

[Detailed Description of the Invention]

[0001] [Field of the Invention]

This invention is used for the automatic sampler for HPLC, and relates to an improvement in the washing of washing a channel where a sample solution circulates.

[0002] [Prior Art]

Fig. 4 shows one example of the construction of a measuring device for a conventional auto-sampler. Based on this figure, a simple explanation is given of the operating principles. First, the six-way valve 1 provides the 6 ports A-F for switching the sample induction route and the sample exit route, and any of the ways for liquid routes shown in the solid or broken line can be selectively

made. The solid line conducts through the liquid route, executing downward migration of the sample absorption tube 2, with insertion of the needle 3 established at the tip to within the sample liquid 5 which was filled in the sample bottle 4. Next, drawing out the piston of the measuring syringe 6, a specified quantity of the sample solution 5 is absorbed from the needle 3.

[0003] Next, The broken line shows the condition of switching the liquid route of the six-way valve. Under these conditions, only the specified amount of the sample liquid is injected into the sample loop 7 by pushing the piston of the measuring syringe 6.

[0004] Afterwards, by switching the liquid route once again of the six-way valve the conditions reverts to the original solid line. The eluted liquid 10 within the tank 9 is sends out to column 11 the sample within the sample loop by mans of the pump 8. The sample is detected by the detector 12, separating every component within the column 11.

[0005] If one measurement cycle is complete, washing is performed within each liquid route by providing measurement for the next time. Here, first, washing occurs using the eluted liquid 10 within the liquid route through which the eluted liquid 10 has passed having pushed out the sample liquid 5. However, the path through which the eluted liquid 10 does not pass, that is, for example, the needle 3 and the sample liquid absorption tube 2 are not washed by the eluted liquid 10. Conventionally, with conditions shown by the broken line

for switching the liquid path with a three-way valve 13, after absorbing the washing liquid 15 which filled within the washing liquid column 14 within the measuring syringe 6 by pulling on the piston of the measuring syringe 6, returns to the original solid line condition by switching the liquid route of the three-way valve 13. The washing liquid 15 within the measuring syringe 6, by pushing the piston is supplied to the needle 3 by means of the sample liquid absorption tube 2. That is, the washing liquid 15 is made to flow in the opposite direction from that when absorbing the sample liquid. By this reverse flow, the interior of the sample liquid injection tube 2 and needle 3 are washed.

[0006] However, the following problems exist with the described conventional washing method. That is, when the needle 3 is absorbing the sample liquid, in order to insert the needle's tip in the sample liquid 5, the sample liquid 5 is attached to the outside surface of the needle 3. Consequently, washing of some sample liquid 5 must also be done. However, using the conventional method, with the main goal to wash the internal liquid route exclusively by passing the washing liquid 15 through the sample liquid absorption tube 2, it is not possible to completely wash the exterior surface of the related needle 3.

[0007] In addition, when the sample liquid 5 is a sample from the body, such as blood serum or urine, protein and lipids adhere to the Teflon (trademark) forming each liquid path for the sample liquid

absorption tube 2. In order to eliminate the adhered protein, it is necessary to wash using alcohol, acid and every kind of organic solvent. However, with conventional devices, because there is only a single washing liquid column 14, the washing liquid 15 which can be supplied to the needle 3 when washing became of a single kind. Consequently, in response to the type of sample liquid 5 to be measured, because one kind is selected to give the best effect among the 3 kinds of washing liquids mentioned above, there is concern that a sufficient washing effect will not be demonstrated. That is, when washing liquid with alkali properties is used, it is not possible to eliminate substances having acidic or organic solvent properties, and there is concern that substances adhered to the inner wall surfaces within the flow path will remain.

[0008] This invention is aware of the previously stated facts and technical experience, and has as its goal to be able to accurately measure by washing the interior flow path through which flows the sample liquid, and furthermore to provide an auto-sampler flow path washing method and an auto-sampler which control contaminant generation.

[0009] This invention is a method for washing the channel of an auto-sampler wherein a needle which has absorbed the sample solution established at the tip of the trial liquid absorption tube is inserted into the washing liquid, absorbing out this washing liquid

by approximately the same operation as absorbing in the trial liquid to the sample liquid absorbing in tube by means of the needle, and next, inserting the needle into a washing port providing a discharge outlet, and under these conditions emitting the washing liquid by making flow the washing liquid in a reverse direction to the absorbing direction to the washing port outside of the needle, and the washing liquid which was emitted, by absorption processing, is discarded forcibly from the discharge outlet (Claim 1). In addition, the invention is a method for washing the channel of an auto-sampler wherein a plurality of different washing liquids are prepared, and along with sequential insertion of the needle within this plurality of washing liquids the washing liquid is absorbed in and emitted out (Claim 2).

[0010] The auto-sample required to execute the stated method is an auto-sampler having a sample liquid absorbing tube on which a needle is mounted at the tip, a sample bottle which stores the sample liquid for absorption through the needle, and a path which supplies to a column the sample liquid which is absorbed by the needle comprising a washing bottle for storing the washing liquid, a washing port which provides a discharge outlet, and an absorption means connected to the discharge outlet, and after temporarily absorbing the washing liquid stored in the washing bottle by means of the needle which has absorbed the sample liquid, possibly discharging the absorbed washing liquid, together with moving to within the washing

port, within the washing port from the needle, and in addition, absorbing the washing liquid emitted to the washing port from the needle by means of the absorbing means, possibly strongly discharging.

[0011] [Use]

By directly inserting the needle within the washing liquid, the outside surface of the needle touches the washing liquid, and when measuring, washing is performed of the sample liquid attached to the outer surface. The washing liquid is absorbed within the sample liquid absorption tube as is by means of the needle, and next, when emitting the absorbed washing liquid to the outside by reverse flow, washing of the inner liquid path of the sample liquid absorption tube occurs.

[0012] Because it is possible for the absorption of this washing liquid to operate in the same way as with the absorption process for the sample liquid when normally measured, a special construction is not necessary, and it is possible, for example, to wash the liquid path by using various kinds of washing liquid by sequentially inserting the needle into differing kinds of washing liquid.

[0013] [Embodiments]

A more detailed explanation, referencing the attached drawings is given of appropriate embodiments of auto-sample flow path washing methods and auto-samplers related to this invention. Fig. 1 shows one example of an appropriate device which executes Embodiment 1 of this invention. As shown in the figure, approximately the same

construction as that conventionally is adopted as the fundamental construction for a measuring system. That is, by appropriately switching a 6-way valve 20, and by supplying temporarily to a sample loop 24 eluted liquid 27 within the tank 26 by pump 25 after storing the sample liquid 22 within the sample bottle 21 by means of the sample liquid absorption tube 23, pulling on the column 28 supplies to the detector 29 the sample 22 which was temporarily stored.

[0014] The low end absorption inlet 33a of the washing liquid absorption tube 33 is inserted and placed within the washing liquid tank 32 which is filled with the 1st washing liquid 30. The other end of the washing liquid absorption tube 33 is linked to the 1st port a of the 3-way valve 34. On the one hand, the 2nd port b of this 3-way valve 34 is linked to the measuring syringe 35, shown by the broken line in the figure, and by pulling the piston 35a of the measuring syringe 35 when the 1st and 2nd ports a and b of the 3-way valve 34 are linked, the 1st washing liquid 30 is absorbed within the measuring syringe 35.

[0015] In addition, the 3rd port c of the 3-way valve 34 is connected to the port F of the 6-way valve 20 by means of the sample liquid absorption tube 23, and contributes to the absorption of the sample liquid 22 in the same way as conventionally, and to the washing of the inner flow path of the needle 37 which is attached to the sample liquid absorption tube 23 and the tip of the sample absorption tube 23.

[0016] Furthermore, on the sample bottle 21's side, that is, on the needle 37's side, the closed end cylinder-shaped washing port 39 is established for receiving the 1st washing liquid 30 which is emitted from the needle 37. In this example, there is established the discharge water port 39a at the base of this washing port 39, and along with connecting one end of the drain tube 40 to this discharge water port 39a, the other end of the drain tube 40 is connected to the washing liquid storage tank 41. This washing liquid storage tank is connected to the vacuum pump 42.

[0017] Furthermore, there is established in the vicinity of the sample bottle 21, a washing bottle 45 filled with the 2nd washing liquid 44. This 2nd washing liquid 44 may be identical to the 1st washing liquid 30 or may be different, and as necessary, is arbitrarily established. In addition, the washing bottle 45 is established as the sole bottle in this example, though a plurality may be established, and furthermore, there may be used as washing bottles sample bottles (sample bottles) in the same way as sample liquids.

[0018] Using the device with the previously described construction, an explanation is give for the washing method of the liquid path related to Embodiment 1 of this invention. Measurements of the normal sample liquid 22 are completed. By moving the needle 37 in a specified direction, the tip of the needle is inserted and placed within the washing liquid port 39. After the 1st port a and

the 2nd port b are linked as shown by the broken line in Fig. 4 having created the appropriate configuration for the 3-way valve, a specified amount of the 1st washing liquid 30 is absorbed within the measuring syringe 35 by drawing a specified amount of the piston 35a of the measuring syringe 35.

[0019] Next, the flow path of the 3-way valve 35 is switched, linking the 2nd port b and the 3rd port c. In this condition, by pushing the piston 35a of the measuring syringe 35, the 1st washing liquid 30 which was absorbed within the measuring syringe 35 is emitted to within the washing port 39 from the lower end of the needle 37 through the sample liquid absorption tube 23, between the port A and port F of the 6-way valve 20, and the needle 37. By this flow, washing of every inner flow path is performed. In addition, in this example, when 1st the washing liquid 30 which was emitted to within the washing port 39, there is operation of the vacuum pump 42 and absorption, and sending of the 1st washing liquid 30 which was emitted to within the washing port 39 to the tank 42 which stores the washing liquid normally by means of the water discharge port 39a and drain tube 40, and by doing in such a way that the 1st washing liquid 30 whose use is completed within the washing port 39 does not accumulate, adhesion is prevented on the outside surface of the needle 37 of washing liquid that is no longer needed, and no necessity exists for operation.

[0020] In this way, if washing of the normal flow path is completed, next is the washing step related to this invention. That is, first, as shown in Fig. 2, along with insertion of the needle 37 within the washing liquid 44 (same composition as the 1st washing liquid 30 in this example) which filled the washing bottle 45, the 2nd and 3rd ports b and c are linked as shown by the solid line in Fig. 1 through the 3-way valve 34. In this condition, by drawing on the piston 35a of the measuring syringe 35, a specified amount of the 2nd washing liquid 44 is absorbed into the sample liquid absorption tube 23.

[0021] Through insertion in the 2nd washing liquid 44 of the needle 37, and with the outside surface of the needle 37 touching the 2nd washing liquid 44, the sample liquid adhered to the outside surface is washed. Moreover, desirably, before absorption of the 2nd washing liquid 44, more concretely, by drawing the piston 35a of the measuring syringe 35 before insertion of the needle 37 in the 2nd washing liquid 44, a small amount of air is absorbed.

[0022] Next, the needle is moved upward, and the lower end is made to separate from the liquid surface of the 2nd washing liquid 44, and in this condition, by drawing the piston 35a of the measuring syringe 36, a small amount of air is absorbed (reference 4(B)). Positioning is made within the washing port 39 by moving the needle 37. In this condition, by pushing the piston 35a of the measuring syringe 35, the 2nd washing liquid 44 which was temporarily absorbed

is emitted in the direction of the discharge port 39 by the needle 37. The emitted 2nd washing liquid 44 is discharged to the outside in the same way as the 1st washing liquid 30.

[0023] If the washing is completed, along with halting the vacuum pump 42, there is separation from the washing port 39 by raising the needle 37. In this example as previously stated, in order that each of the washing liquids 30 and 40 that are only used within the washing port 39 do not accumulate, when raising and moving the needle, no re-adhesion of the washing liquid on the outside surface of the needle 37 occurs.

[0024] Moreover, there may be a plurality of repeated operations for washing by absorbing and drawing of the needle 37 as necessary, and after a certain time, the washing effect improves. In addition, as with this embodiment, the washing process by absorbing and drawing from the needle 37 that is related to this invention can be for every fixed period or randomly without measurement. Furthermore, in the previously described embodiment it is good to proceed with washing in a reverse sequence after washing in the normal way for washing related to this invention.

[0025] Furthermore, for operations timing of the vacuum pump 42, it is not necessary to perform before penetrating with a needle each of the washing liquids 30 and 44 as in the embodiment, and with all the washing processes using the needle 37 completed, the needle 37 operates before the uplifting movement, and it is good if there can

be discharge of the washing liquid within the washing port 39, and preferably, the washing liquid that is only used within the washing port 39 has accumulated, and before the washing liquid only used at the lower end of the needle 37 makes contact, operation of the back pump 42 is performed.

[0026] Fig. 3 shows the necessary parts of the operational processes for Embodiment 2 of this invention. In this example also, along with insertion of the needle 37 in the 2nd washing liquid 44, there is the process of absorption, and then the process of absorbing a small amount of air after moving the needle 37 upward is done in the same way as with Embodiment 1. Fig. 3(A) shows the conditions of the sample liquid absorption tube 23 after absorbing and drawing some related air and the conditions within the 6-way valve 20. That is, the 2nd washing liquid 44 indicated by black coating is positioned at the center of the sample liquid absorption tube 23 which connects the port F of the 6-way valve 20 and the measuring syringe 35. Moreover, the symbol 47 in the figure is air that was absorbed before absorbing and drawing the 2nd washing liquid 44.

[0027]In these conditions, the piston 35a of the measuring syringe 35 is pushed only a certain amount. The amount pushed is small compared to the amount pulled in the previous process. As shown in Fig. 3(B), the 2nd washing liquid 44 of only a specified amount is pushed and returned. However, as abbreviated in the figure, with air that has been absorbed after absorption of the 2nd washing liquid 44

under these conditions, the 2nd washing liquid 44 does not accumulate from the tip of the needle 37.

[0028] By pushing a specified number of times the piston 35a and drawing it, the conditions of Fig. 3(A) and Fig. 3(B) are repeated. From this repetition the 2nd washing liquid 44 returns and moves within the sample liquid absorption tube 23, and an increased washing effect obtained.

[0029] Afterwards, the needle 37 moves to the washing port 39, and the 2nd washing liquid 44 which was absorbed by pushing a great deal the piston 35a is emitted. Other constructions and operational effects are not explained as they are the same as with Embodiment 1.

[0030] Moreover, with every embodiment described, all the washing liquids that were absorbed by the needle 37 were explained for one kind, but this invention is no so limited, and with the preparation of a plurality of washing bottles, differing kinds in each of the washing bottles, and by filling with washing liquid such as alkali acid or organic solvents good effective washing can occur with the desired washing liquid. By making related constructions, it is possible to wash accurately with sample liquid so as not to be able to completely wash when not using a plurality of washing liquids such as living samples (blood serum, etc.).

[0031] When it is necessary to have 2 kinds of washing liquid, there is not preparation of a plurality of washing bottles as described above, and there is filling of one washing liquid within

the washing liquid tank performing normal washing, and it is possible to obtain the same effect as filling other washing liquid in the washing bottle.

[0032] In addition, in every embodiment discussed, there was an explanation of an example which executed this invention's washing method by adding to all washing methods, and it is permissible to perform washing with a flow path using only the washing method of this invention. In related cases, a 3-way valve and washing liquid tank are not necessary, and it is possible to directly connect the measuring syringe with one of the ports of the 6-waqy valve. Also, simplification of the device was attempted in order to execute this invention's methods.

[0034] [Effect of the Invention]

As stated above, with the flow path washing method of the auto-sampler related to this invention, because the needle is inserted directly in the washing liquid, the outside surface of the needle contacts the washing liquid, and it is possible to wash this outside surface. The washing liquid is absorbed within the sample liquid absorption tube by means of the needle under unchanged conditions and because the absorbed washing liquid is emitted to the outside by reverse flow, it is possible to wash the inner flow path of the sample liquid absorption tube. That is, with this invention, because it is possible to execute the same operation of washing as the absorption process for the sample liquid in normal measurement time,

an effect is also achieved of being able to use as is conventional devices with special devices or constructions.

[0034] Furthermore, it is possible to wash the flow paths by using various kinds of washing liquid by sequentially inserting various different kinds of washing liquid within the needle, and it is possible to wash the flow paths more accurately. Consequently, it is possible to perform accurately the washing process also after measurement of sample liquids which could not perform washing completely if the washing liquids of various kinds for blood serum and urine were not used. The effect, because there is no absorption on Teflon (trademark) which the protein and lipids within the blood serum and urine form every flow path for sample liquid absorption tubes, the volume (cross-sectional area) is normally maintained at a constant, and the sample liquid which is subject to measurement and the sample liquid previously processed are not mixed together, thus giving conditions for accurate measurement. Furthermore, it is possible to perform measurements continuously for the required sample liquid of differing washing liquids, thus improving the operational efficiency.

[Brief Explanation of the Drawings]

[Figure 1] A construction diagram which shows one example of the device which is appropriate for executing Embodiment 1 of the auto-sampler flow path washing method related to this invention.

[Figure 2] A diagram for explaining the use of Embodiment 1.

[Figure 3] A diagram which explains the principal parts of the process of Embodiment 2 of the auto-sampler flow path washing method related to this invention.

[Figure 4] A construction diagram showing a conventional example.

[Explanation of the Elements]

22- sample liquid

23- sample liquid absorption tube

37- needle

44- 2nd washing liquid (washing liquid)

Figure 1

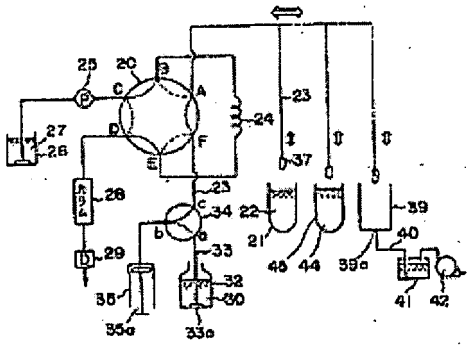
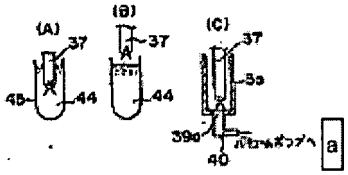


Figure 2



Key: a) To vacuum pump

Figure 3

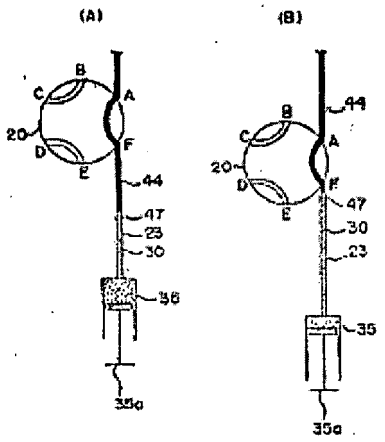
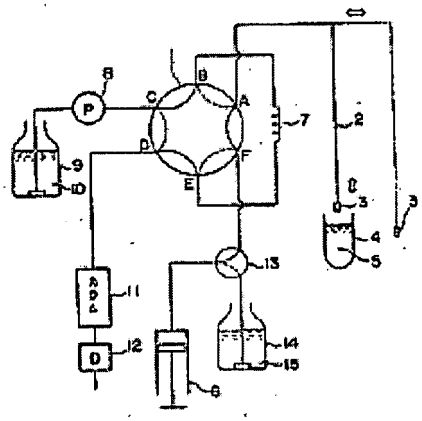


Figure 4



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最終頁に続く

(54)【発明の名称】オートサンプラーの流路洗浄方法及びオートサンプラー

(57)【特許請求の範囲】
【請求項１】　試料液吸入管の先端に設けられた試料液を吸入したニードルを洗浄液内に挿入し、試料液の吸入と略同一作業によりその洗浄液を前記ニードルを介して前記試料液吸入管内に吸引し、
次いで、前記ニードルを、排出口を備えた洗浄ポートに挿入し、その状態で前記吸引方向と逆方向に前記洗浄液を流通させてその洗浄液を前記ニードルより外部の前記洗浄ポート内に吐出し、
前記吐出された前記洗浄液は、吸引処理によって、前記排出口から強制的に廃棄することを特徴とするオートサンプラーの流路洗浄方法。
【請求項２】　異なる複数の洗浄液を用意し、前記ニードルをその複数の洗浄液内に順次挿入するとともにその洗浄液を吸引し、吐出することを特徴とする請求項１に

記載のオートサンプラーの流路洗浄方法。
【請求項３】　先端にニードルが取り付けられた試料液吸入管と、
前記ニードルを介して吸入するための試料液を貯留する試料瓶と、
前記ニードルを介して吸入された試料液を、カラムに供給する経路を備えたオートサンプラーにおいて、
洗浄液を貯留するための洗浄瓶と、排出口を備えた洗浄ポートと、前記排出口に接続された吸引手段とを備え、
前記洗浄瓶内に貯留された前記洗浄液を試料液を吸入した前記ニードルを介して一旦吸入後、前記ニードルの先端を前記洗浄ポート内に移動させるとともにその吸入した前記洗浄液を前記ニードルから前記洗浄ポート内に排出可能とし、かつ、前記吸引手段にて前記ニードルから前記洗浄ポートに吐出された前記洗浄液を吸引し、強制

的に廃棄可能としたことを特徴とするオートサンプラー。

【発明の詳細な説明】

【０００１】

【産業上の利用分野】本発明はＨＰＬＣ用のオートサンプラーに用いられ、試料液が流通する流路を洗浄する方法の改良に関する。

【０００２】

【従来の技術】図４に従来のオートサンプラーの測定装置の構成例を一例を示している。同図に基づいてその動作原理を簡単に説明する。まず、６個のポートＡ～Ｆを備えた六方弁１は、試料導入ルートと試料導出ルートとを切り替えるためのものであり、実線で示す流路と破線で示す流路のいずれか一方が選択的に導通される。この実線で示す流路を導通させた状態で、試料吸入管２を下降移動させて、その先端に設けられたニードル３を試料瓶４内に充填された試料液５内に挿入する。次いで、計量シリンジ６のピストンをひいて、ニードル３から試料液５を所定量吸引する。

【０００３】次いで、六方弁１の流路を切り替えて破線に示す状態とする。この状態で計量シリンジ６のピストンを押して所定量だけサンプルループ７内に試料液を注入する。

【０００４】その後、再び六方弁１の流路を切り替えて、元の実線に示す状態にする。そして、ポンプ８によって貯槽９内の溶離液１０がサンプルループ内の試料をカラム１１へ送出する。試料はカラム１１内で各成分に分離され検出器１２で検知される。

【０００５】そして、一回の測定が終了したなら、次回の測定に備えて各流路内の洗浄を行う。ここでまず上記の試料液５の押し出しの際に溶離液１０が通過した流路内はその溶離液１０で洗浄される。しかし、溶離液１０の通過しない経路、すなわち、ニードル３並びに試料液吸入管２等は溶離液１０では洗浄されない。そこで従来は、三方弁１３の流路を切り替えて破線に示す状態とし、その状態で計量シリンジ６のピストンを引いて計量シリンジ６内に洗浄液貯槽１４内に充填された洗浄液１５を吸入させた後、三方弁１３の流路を切り替えて元の実線の状態に戻す。そして、ピストンを押すことにより計量シリンジ６内の洗浄液１５を試料液吸入管２を介してニードル３側へ供給し、そのニードル３より外部へ吐出させる。すなわち、上記の試料液の吸入と逆方向に洗浄液１５を流すことになる。これにより試料液注入管２並びにニードル３等の内部が洗浄される。

【０００６】

【発明が解決しようとする課題】しかしながら、上記した従来の洗浄方法では、以下に示す問題がある。すなわち、ニードル３は試料液吸入時にその先端を試料液５内に挿入させているため、ニードル３の外側表面にも試料液５が付着している。したがって、かかる試料液５の洗浄も行わなければならない。しかし従来の洗浄方法では洗浄液１５を試料液吸入管２内を通過させているというように専らその内部流路の洗浄に主目的をおいており、係るニードル３の外側表面を充分に洗浄することはできない。

【０００７】また、上記試料液５が生体サンプル、特に血清及び尿等の場合には、その試料液５中の蛋白質及び脂質が試料液吸入管２等の各流路を形成するテフロン（登録商標）に吸着する。そして、それら吸着した蛋白質等を除去するためには、アルカリ，酸，有機溶媒各種による洗浄が必要となる。しかし、上記した従来の装置では、単一の洗浄液貯槽１４しか有していないため、洗浄時にニードル３側へ供給できる洗浄液１５は一種類となる。したがって、測定する試料液５の種類に応じて、上記３種類の洗浄液のうち最も洗浄効果のあがる一種を選択し使用していたため、充分な洗浄効果が発揮し得ないおそれがある。すなわち、仮にアルカリ性の洗浄液を用いた場合には、酸性や有機溶媒の洗浄液で落ちる物質が除去できずに流路内壁面に付着したままになるおそれがある。

【０００８】本発明は上記した背景に鑑みてなされたもので、その目的とするところは、ニードルの外側表面や、試料液の流れる内部流路等を確実に洗浄することにより、正確な測定を行うことができ、しかも汚染の発生を抑制するオートサンプラーの流路洗浄方法及びオートサンプラーを提供することにある。

【０００９】

【課題を解決するための手段】上記した目的を達成するために、本発明に係るオートサンプラーの流路洗浄方法では、試料液吸入管の先端に設けられた試料液を吸入したニードルを洗浄液内に挿入し、試料液の吸入と略同一作業によりその洗浄液を前記ニードルを介して前記試料液吸入管内に吸引し、次いで、、前記ニードルを、排出口を備えた洗浄ポートに挿入し、その状態で前記吸引方向と逆方向に前記洗浄液を流通させてその洗浄液を前記ニードルより外部の前記洗浄ポート内に吐出し、前記吐出された前記洗浄液は、吸引処理によって、前記排出口から強制的に廃棄するようにした（請求項１）。また、好ましくは異なる複数の洗浄液を用意し、前記ニードルをその複数の洗浄液内に順次挿入するとともにその洗浄液を吸引し、吐出することである（請求項２）。

【００１０】そして、上記方法を実施するためのオートサンプラーとしては、先端にニードルが取り付けられた試料液吸入管と、前記ニードルを介して吸入するための試料を貯留する試料瓶と、前記ニードルを介して吸入された試料液を、カラムに供給する経路を備えたオートサンプラーにおいて、洗浄液を貯留するための洗浄瓶と、排出口を備えた洗浄ポートと、前記排出口に接続された吸引手段とを備え、前記洗浄瓶内に貯留された前記洗浄液を試料液を吸入した前記ニードルを介して一旦吸入

後、前記ニードルの先端を前記洗浄ポート内に移動させるとともにその吸入した前記洗浄液を前記ニードルから前記洗浄ポート内に排出可能とし、かつ、前記吸引手段にて前記ニードルから前記洗浄ポートに吐出された前記洗浄液を吸引し、強制的に廃棄可能に構成することができる（請求項３）。

【００１１】

【作用】ニードルを洗浄液内に直接挿入させることにより、そのニードルの外側表面が洗浄液に触れ、測定時にその外側表面に付着した試料液の洗浄が行われる。そして、その状態のままニードルを介して試料液吸入管内に洗浄液を吸引し、次いで、その吸引した洗浄液を逆流させて外部に吐出させると、試料液吸入管の内部流路の洗浄が行われる。

【００１２】そして、この洗浄液の吸入は、通常の測定時における試料液の吸入工程と同一の作業で行うことができるので、特殊な機構は不要であり、例えばニードルを異なる種類の洗浄液内に順次挿入することにより複数種の洗浄液を用いて流路の洗浄を行うことができる。

【００１３】

【実施例】以下本発明に係るオートサンプラーの流路洗浄方法及びオートサンプラーの好適な実施例を添付図面を参照にして詳述する。図１は本発明の第１実施例を実施するに適した装置の一例を示している。同図に示すように、測定系の基本構成は上記した従来のものと略同一構成を採っている。すなわち、六方弁２０を適宜切替えることにより、試料瓶２１内の試料液２２を試料液吸入管２３を介して一旦サンプルループ２４内に貯留させた後、ポンプ２５によって貯槽２６内の溶離液２７をサンプルループ２４側に供給することにより、上記一旦貯留されていた試料液２２をカラム２８ひいては検出器２９に供給するようにしている。

【００１４】そして、第１の洗浄液３０が充填されている洗浄液貯槽３２内に洗浄液吸入管３３の下端吸入口３３ａを挿入配置し、その洗浄液吸入管３３の他方端部を三方弁３４の第１のポートａに連結する。一方、この三方弁３４の第２のポートｂは、計量シリンジ３５に連繋されており、図中破線で示す如く、三方弁３４の第１，第２のポートａ，ｂが連通された状態で計量シリンジ３５のピストン３５ａを引くことにより、第１の洗浄液３０が計量シリンジ３５内に吸引される。

【００１５】また、三方弁３４の第３のポートｃは、試料液吸引管２３を介して六方弁２０のポートＦに接続されており、従来と同様試料液２２の吸引や、試料液吸引管２３並びに試料液吸引管２３の先端に装着されたニードル３７の内側流路の洗浄に寄与される。

【００１６】さらにまた、試料瓶２１側、すなわちニードル３７側には、ニードル３７から吐出される第１の洗浄液３０を受けるための有底円筒状の洗浄ポート３９が配設されている。そして本例ではその洗浄ポート３９の

底部に排水口３９ａを設け、その排水口３９ａにドレイン管４０の一端を接続すると共に、ドレイン管４０の他端を洗浄液溜めタンク４１に連通させる。そして、その洗浄液溜めタンク４１は、バキュームポンプ４２に連繋させている。

【００１７】さらに試料瓶２１の近傍に、第２の洗浄液４４が充填された洗浄瓶４５が設けられている。この第２の洗浄液４４は、上記した第１の洗浄液３０と同一のものでも良く、或いは成分の異なるものでも良く、必要に応じて任意に設定される。また、洗浄瓶４５は、本例では１個設けているが、複数個設けても良く、さらに、試料液と同様にサンプル瓶（試料瓶）を洗浄瓶として使用するようにしてもよい。

【００１８】上記構成の装置を用いて、本発明の第１実施例に係る流路の洗浄方法について説明する。今、通常の試料液２２の測定が終了したとする。すると、ニードル３７を所定方向に移動させて、そのニードル３７の先端を洗浄ポート３９内に挿入配置させる。そして、三方弁３４を図示の破線で示すように第１のポートａと第２のポートｂとが連通する状態にした後、計量シリンジ３５のピストン３５ａを所定量引くことにより計量シリンジ３５内に第１の洗浄液３０を所定量吸入する。

【００１９】次いで、三方弁３４の流路を切り替え、第２のポートｂと第３のポートｃとが連通する状態にする。この状態で、計量シリンジ３５のピストン３５ａを押すことにより、計量シリンジ３５内に吸入された第１の洗浄液３０は、試料液吸入管２３，六方弁２０のポートＡ，Ｆ間並びにニードル３７を通ってニードル３７の下端より洗浄ポート３９内に向けて吐出される。これにより各内側流路の洗浄が行われる。また、本例では、このニードル３７から第１の洗浄液３０が吐出されている間、バキュームポンプ４２を作動させて吸引し、洗浄ポート３９内に吐出された第１の洗浄液３０を常時排水口３９ａ，ドレイン管４０を介して洗浄液溜めタンク４２側に送り、洗浄ポート３９内にはその使用済みの第１の洗浄液３０が溜まらないようにし、使用済み洗浄液のニードル３７の外側表面への再付着を防止しているが、必ずしも動作させる必要はない。

【００２０】このようにして通常の流路の洗浄が終了したなら、本発明に係る洗浄のステップに移る。すなわち、まず図２（Ａ）に示すようにニードル３７を洗浄瓶４５内に充填されている第２の洗浄液４４（本例では第１の洗浄液３０と同一組成）内に挿入するとともに、三方弁３４を図１中実線で示すように第２，第３のポートｂ，ｃ間を連通状態とする。この状態で計量シリンジ３５のピストン３５ａを引くことにより、所定量の第２の洗浄液４４を試料液吸入管２３内に吸入する。

【００２１】そして、ニードル３７の第２の洗浄液４４内への挿入にともない、ニードル３７の外側表面が第２の洗浄液４４に触れることになり、その外側表面に付着

した試料液が洗浄される。尚、好ましくは上記第２の洗浄液４４を吸引する前、より具体的にはニードル３７が第２の洗浄液４４内に挿入する前に計量シリンジ３５のピストン３５ａを引くことにより、少量の空気を吸引させておくことである。

【００２２】次いで、ニードル３７を上昇移動させ、その下端を第２の洗浄液４４の液面より離反させ、この状態でさらに計量シリンジ３５のピストン３５ａを引くことにより少量の空気を吸い込む（同図（Ｂ）参照）。そして、ニードル３７を移動させて洗浄ポート３９内に位置させる。この状態で計量シリンジ３５のピストン３５ａを押すことにより一旦吸引した第２の洗浄液４４をニードル３７より排出ポート３９に向けて吐出させ（同図（Ｃ）参照）、内側流路の洗浄が終了する。そして、この吐出された第２の洗浄液４４は、上記第１の洗浄液３０と同様に外部へ排出される。

【００２３】そして、上記洗浄が終了したなら、バキュームポンプ４２を停止すると共に、ニードル３７を上昇させ洗浄ポート３９より離反させる。上述したごとく本例では洗浄ポート３９内に使用済みの各洗浄液３０，４４が貯留していないため、ニードル３７の上昇移動の際に、ニードル３７の外側表面に洗浄液が再付着するようなことはない。

【００２４】尚、必要に応じて上記のニードル３７からの吸引による洗浄処理を複数回繰り返して行うようにしても良く、かかる場合にはより洗浄効果が上がる。また、本実施例のように、通常の洗浄処理と併用する場合には、本発明に係るニードル３７からの吸引による洗浄処理は、測定の都度行わずに一定間隔毎、或いはランダム的に行うようにしても良い。さらに、上記実施例では、通常の洗浄をした後に本発明に係る洗浄を行うようにしたが、この順番は逆でも良い。

【００２５】さらには、バキュームポンプ４２の作動タイミングは、上述した実施例の如く各洗浄液３０，４４をニードルより噴射する前から行う必要はなく、ニードル３７に対するすべての洗浄工程が終了し、ニードル３７を上昇移動させるより前に作動させ、洗浄ポート３９内の洗浄液を排出できれば良いが、好ましくは、洗浄ポート３９内に使用済みの洗浄液が溜まり、ニードル３７の下端に使用済みの洗浄液が接触する前にバキュームポンプ４２を作動させることである。

【００２６】図３は本発明の第２実施例の作業工程の要部を示している。本例でもニードル３７を第２の洗浄液４４内に挿入すると共に吸引し、次いでニードル３７を上昇移動させた後少量の空気を吸引する工程は、上記第１実施例と同様である。そして、係る空気を吸引した後の試料液吸入管２３並びに六方弁２０内の状態を示すと図３（Ａ）に示すようになっている。すなわち、黒塗りして示す第２の洗浄液４４は、六方弁２０のポートＦと計量シリンジ３５とを結ぶ試料液吸入管２３の中央部位

まで位置している。尚、図中符合４７は、第２の洗浄液４４の吸引前に吸引した空気である。

【００２７】この状態で、計量シリンジ３５のピストン３５ａを所定量だけ押す。この押す量は、前工程で引いた量よりも少なくしてる。すると、同図（Ｂ）に示すように、所定量だけ第２の洗浄液４４が押し戻される。但し、図示省略するがこの状態においても第２の洗浄液４４の吸引後に吸引した空気により、ニードル３７の先端より第２の洗浄液４４がもれ出ることはない。

【００２８】そして、上記ピストン３５ａを所定回数だけ押し・引き操作することにより、図３（Ａ）と（Ｂ）の状態を繰り返す。これにより、第２の洗浄液４４が試料液吸入管２３内を往復移動することになり、より洗浄効果が増す。

【００２９】その後、ニードル３７を洗浄ポート３９側へ移動し、ピストン３５ａを大きく押し込むことにより吸入した第２の洗浄液４４を排出する。その他の構成並びに作用効果は上記した第１実施例と同様であるのでその説明を省略する。

【００３０】尚、上記した各実施例では、いずれもニードル３７を介して吸引する洗浄液は一種類のものについて説明したが、本発明はこれに限ることはなく、複数の洗浄瓶を用意し、各洗浄瓶内に異なる種類、例えばアルカリ性，酸性，有機溶媒等の洗浄液を充填しておき、所望の洗浄液に対して、上記の洗浄作業を行うようにしても良い。係る構成にすることにより、例えば生体サンプル（血清など）のように複数種の洗浄液を用いないと完全に洗浄することができないような試料液に対しても確実に洗浄処理を行うことができる。

【００３１】そして、係る複数種、特に２種類の洗浄液が必要な場合には、上記のように複数の洗浄瓶を用意することなく、通常の洗浄を行う洗浄液貯槽内に一方の洗浄液を充填しておき、洗浄瓶内に他方の洗浄液を充填することにより同様の効果を得ることができる。

【００３２】尚また、上記した各実施例ではいずれも通常の洗浄方法に加えて本発明の洗浄方法を実施する例について説明したが、本発明に係る洗浄方法のみで流路等の洗浄を行うようにしても良い。係る場合には、三方弁並びに洗浄液貯槽が不要となり、計量シリンジを六方弁の一のポートに直接接続することができ、本発明方法を実施するための装置の簡略化が図れる。

【００３３】

【発明の効果】以上のように、本発明に係るオートサンプラーの流路洗浄方法では、ニードルを直接洗浄液内に挿入するため、ニードルの外側表面が洗浄液に接触し、これによりその外側表面を洗浄することができる。そしてその状態のままニードルを介して試料液吸入管内に洗浄液を吸引し、次いで、その吸引した洗浄液を逆流させて外部に吐出させるため、試料液吸入管の内部流路の洗浄も行うことができる。すなわち、本発明では、洗浄作

業を通常の測定時における試料液の吸入工程と同一の作業で行えることができるので、特殊な装置・機構は不要で従来の装置をそのまま使用することができるという効果も奏する。

【００３４】しかも、例えばニードルを異なる種類の洗浄液内に順次挿入することにより複数種の洗浄液を用いて流路の洗浄を行うことができ、より確実に流路を洗浄することができる。したがって、例えば血清や尿等の複数種の洗浄液を用いなければ完全に洗浄を行うことができない試料液の測定後であっても確実に洗浄処理を行うことができる。その結果、係る血清及び尿等の中の蛋白質及び脂質が試料液吸入管等の各流路を形成するテフロン（登録商標）に吸着することがないため、流路内の容積（断面積）は常に一定に保たれるとともに、測定対象の試料液と前回行った試料液とが混在したりすることもなく、正確な測定を行うことができる。さらに、異なる

洗浄液の必要な試料液に対しても連続して測定を行うことができ、作業効率が向上する。

【図面の簡単な説明】

【図１】本発明に係るオートサンプラーの流路洗浄方法の第１実施例を実施するに適した装置の一例を示す構成図である。

【図２】第１実施例の作用を説明するための図である。

【図３】本発明に係るオートサンプラーの流路洗浄方法の第２実施例の工程の要部を示す図である。

【図４】従来例を示す構成図である。

【符号の説明】

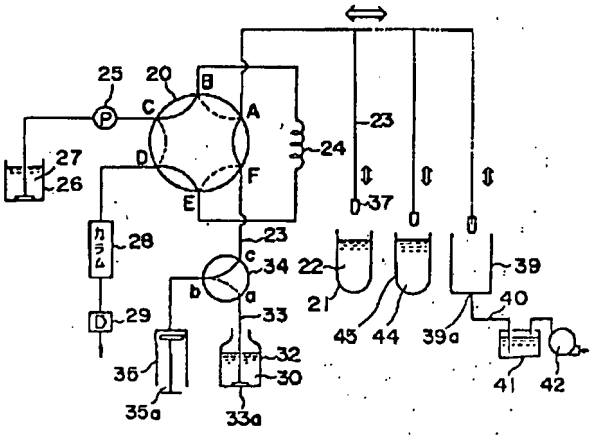
２２　試料液

２３　試料液吸入管

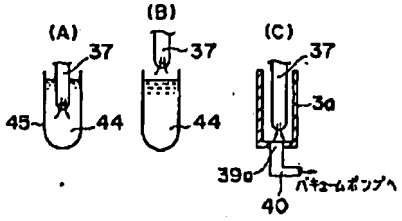
３７　ニードル

４４　第２の洗浄液（洗浄液）

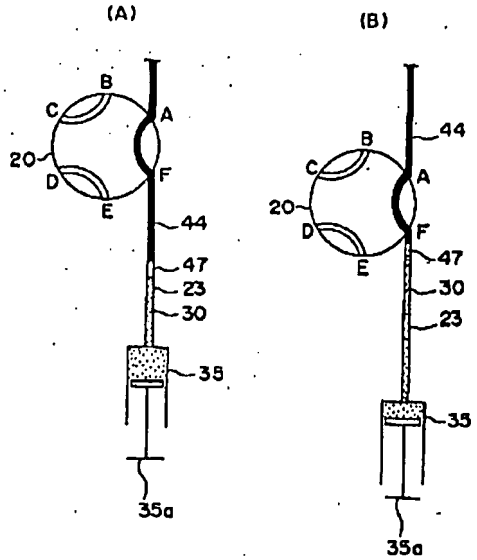
【図１】



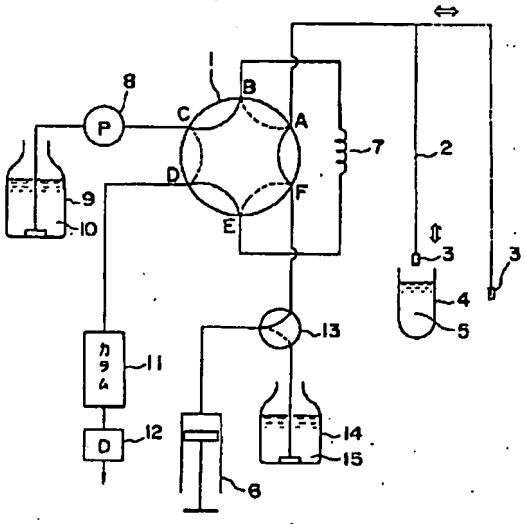
【図２】



【図３】



【図４】

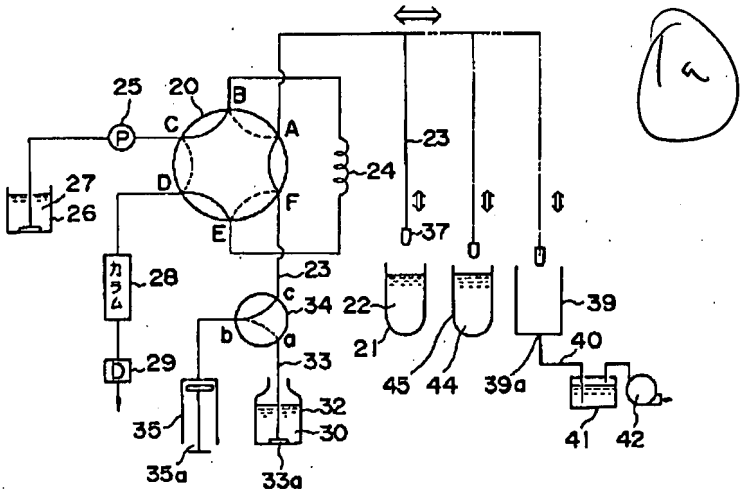


フロントページの続き

(56)参考文献　特開 昭63－158464（ＪＰ，Ａ）

(58)調査した分野(Int.Cl.⁷，ＤＢ名)
G01N 30/24
G01N 1/00

Drawing selection drawing 1



[Translation done.]

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1.This document has been translated by computer. So the translation may not reflect the original precisely.
2.**** shows the word which can not be translated.
3.In the drawings, any words are not translated.

DETAILED DESCRIPTION

[Detailed Description of the Invention]
[0001]
[Industrial Application] This invention is used for the automatic sampler for HPLC, and relates to amelioration of the approach of washing the passage where a sample solution circulates.
[0002]
[Description of the Prior Art] An example is shown for the example of a configuration of the measuring device of the conventional automatic sampler in drawing 4 . Based on this drawing, the principle of operation is explained briefly. First, either of the passage shown with the passage which it is and is shown as a continuous line and the broken line for changing the sample installation root and the sample derivation root flows through the roppo valve 1 equipped with six port A-F alternatively. In the condition of having made it flowing through the passage shown as this continuous line, downward migration of the sample suction pipe 2 is carried out, and it inserts into the sample solution 5 filled up with the needle 3 prepared at that tip in the specimen bottle 4. Subsequently, the piston of the measuring syringe 6 is pulled and specified quantity suction of the sample solution 5 is carried out from a needle 3.
[0003] Subsequently, it considers as the condition which the passage of the roppo valve 1 is changed and shows in a broken line. The piston of the measuring syringe 6 is pushed in this condition, and only the specified quantity pours in a sample solution into the sample loop formation 7.
[0004] Then, the passage of the roppo valve 1 is changed again and it changes into the condition which shows in the original continuous line. And the eluate 10 in a tank 9 sends out the sample within a sample loop formation to a column 11 with a pump 8. It separates into each component within a column 11, and a sample is detected with a detector 12.
[0005] And if one measurement is completed, washing in each passage will be performed in preparation for next measurement. The inside of the passage through which the eluate 10 passed first here on the occasion of extrusion of the above-mentioned sample solution 5 is washed by the eluate 10. However, sample solution suction-pipe 2 grade is not washed by the path which an eluate 10 does not pass, i.e., needle 3 list, by the eluate 10. Then, conventionally, after making the penetrant remover 15 with which considered as the condition which the passage of a cross valve 13 is changed and shows in a broken line, lengthened the piston of the measuring syringe 6 in the condition, and it filled up in the penetrant remover tank 14 in the measuring syringe 6 inhale, the passage of a cross valve 13 is changed and it returns to the condition of the original continuous line. And the penetrant remover 15 in the measuring syringe 6 is supplied to a needle 3 side through the sample solution suction pipe 2, and it is made to breathe out from the needle 3 to the exterior by pushing a piston. That is, a penetrant remover 15 will be passed to inhalation and hard flow of the above-mentioned sample solution. Thereby, the interior of needle 3 grade is washed by sample solution filling pipe 2 list.
[0006]
[Problem(s) to be Solved by the Invention] However, there is a problem shown below by the above-mentioned conventional washing approach. That is, since the needle 3 is making the tip insert into a sample solution 5 at the time of sample solution inhalation, the sample solution 5 has adhered also to the

outside front face of a needle 3. Therefore, washing of this sample solution 5 must also be performed. However, by the conventional washing approach, the key objective is chiefly set to washing of the internal passage, and the outside front face of the starting needle 3 cannot fully be washed as the inside of the sample solution suction pipe 2 is passed for the penetrant remover 15.

[0007] Moreover, in the case of a living body sample especially a blood serum, urine, etc., the above-mentioned sample solution 5 adsorbs at the Teflon (trademark) in which the protein and the lipid in the sample solution 5 form each passage of sample solution suction-pipe 2 grade. And in order to remove the protein to which it they-stuck, washing by alkali, the acid, and organic solvent various kinds is needed. However, with the above-mentioned conventional equipment, since it has only the single penetrant remover tank 14, the penetrant remover 15 which can be supplied to a needle 3 side at the time of washing becomes one kind. Therefore, since it was used according to the class of sample solution 5 to measure, having chosen a kind which a cleaning effect goes up most among the three above-mentioned kinds of penetrant removers, there is a possibility that sufficient cleaning effect cannot demonstrate. That is, when an alkaline penetrant remover is used temporarily, there is a possibility of remaining adhering to a passage internal surface, without the matter which falls off by the penetrant remover of acidity or an organic solvent being unremovable.

[0008] By washing certainly the outside front face of a needle, the internal passage where a sample solution flows, the place which this invention was made in view of the above-mentioned background, and is made into the purpose can perform exact measurement, and is to offer the passage washing approach of the automatic sampler which moreover controls generating of contamination.

[0009]

[Means for Solving the Problem] In order to attain the above-mentioned purpose, the needle prepared at the tip of a sample solution suction pipe was inserted into the penetrant remover, and the penetrant remover was attracted in said sample solution suction pipe through said needle according to inhalation of a sample solution, and an abbreviation same activity, and more nearly subsequently to said suction direction and hard flow than said needle, said penetrant remover is circulated and it was made to carry out the regurgitation of the penetrant remover outside by the passage washing approach of the automatic sampler concerning this invention.

[0010] Moreover, while preparing two or more preferably different penetrant removers and carrying out sequential insertion of said needle into two or more of the penetrant removers, it is attracting and carrying out the regurgitation of the penetrant remover.

[0011]

[Function] By making a needle insert directly into a penetrant remover, the outside front face of the needle touches a penetrant remover, and washing of the sample solution which adhered to the outside front face at the time of measurement is performed. And a penetrant remover is attracted in a sample solution suction pipe through a needle with the condition, and if the attracted penetrant remover is made to flow backwards and it is subsequently made to breathe out outside, washing of the internal passage of a sample solution suction pipe will be performed.

[0012] And since inhalation of this penetrant remover can be performed by the same activity as the inhalation process of the sample solution at the time of the usual measurement, the special device is unnecessary, for example, can wash two or more passage using the penetrant remover of a seed by carrying out sequential insertion of the needle into the penetrant remover of a different class.

[0013]

[Example] An accompanying drawing is made reference and the suitable example of the passage washing approach of the automatic sampler which starts this invention below is explained in full detail. Drawing 1 shows an example of the equipment suitable for carrying out the 1st example of this invention. As shown in this drawing, the basic configuration of system of measurement has taken the above-mentioned conventional thing and the above-mentioned conventional abbreviation same configuration. That is, after making the sample solution 22 in a specimen bottle 21 once store in the sample loop formation 24 through the sample solution suction pipe 23 by changing the roppo valve 20 suitably, he is trying to supply the sample solution 22 ******(ed) [above-mentioned] one to a column

28, as a result a detector 29 by supplying the eluate 27 in a tank 26 to the sample loop-formation 24 side with a pump 25.

[0014] And insertion arrangement of the lower limit inhalation opening 33a of the penetrant remover suction pipe 33 is carried out into the penetrant remover tank 32 with which it fills up with the 1st penetrant remover 30, and the another side edge of the penetrant remover suction pipe 33 is connected with the 1st port a of a cross valve 34. On the other hand, the 2nd port b of this cross valve 34 is coordinated with the measuring syringe 35, and as a drawing destructive line shows, the 1st penetrant remover 30 is attracted in the measuring syringe 35 by lengthening piston 35a of the measuring syringe 35, where the 1st and 2nd port a and b of a cross valve 34 is opened for free passage.

[0015] Moreover, it connects with the port F of the roppo valve 20 through the sample solution siphon 36, and the 3rd port c of a cross valve 34 contributes to suction of a sample solution 22, and washing of the inside passage of the needle 37 with which the sample solution siphon 36 list was equipped at the tip of the sample solution siphon 36 as usual.

[0016] The washing port 39 of the shape of a closed-end cylinder for receiving the 1st penetrant remover 30 breathed out from a needle 37 is arranged in the specimen bottle 21 37, i.e., needle, side further again. And while preparing exhaust-port 39a in the pars basilaris ossis occipitalis of the washing port 39 and connecting the end of a drain pipe 40 to the exhaust-port 39a, the penetrant remover reservoir tank 41 is made to open the other end of a drain pipe 40 for free passage in this example. And the vacuum pump 42 is made to coordinate the penetrant remover reservoir tank 41.

[0017] Furthermore near the specimen bottle 21, the washing bottle 45 with which the 2nd penetrant remover 44 was filled up is formed. It may be the same as that of the 1st above-mentioned penetrant remover 30, or components may differ, and this 2nd penetrant remover 44 is set as arbitration if needed. Moreover, although one washing bottle 45 is formed by this example, it may be prepared and you may make it use a sample bottle (specimen bottle) as a washing bottle like a sample solution further. [two or more]

[0018] The washing approach of the passage concerning the 1st example of this invention is explained using the equipment of the above-mentioned configuration. Now, suppose that measurement of the usual sample solution 22 was completed. Then, a needle 37 is moved in the predetermined direction and insertion arrangement of the tip of the needle 37 is carried out into the washing port 39. And after changing into the condition that the 1st port a and the 2nd port b are open for free passage, as [show / a cross valve 34 / the broken line of illustration], specified quantity inhalation of the 1st penetrant remover 30 is carried out for piston 35a of the measuring syringe 35 into the measuring syringe 35 by specified quantity ******.

[0019] Subsequently, the passage of a cross valve 34 is changed and it changes into the condition that the 2nd port b and the 3rd port c are open for free passage. In this condition, the 1st penetrant remover 30 inhaled in the measuring syringe 35 is breathed out towards the inside of the washing port 39 by pushing piston 35a of the measuring syringe 35 by the port A of the sample solution suction pipe 36 and the roppo valve 20, and the list between F through a needle 37 from the lower limit of a needle 37. Thereby, washing of each inside passage is performed. Moreover, while the 1st penetrant remover 30 is breathed out from this needle 37 in this example, A vacuum pump 42 is operated, it draws in and exhaust-port 39a and a drain pipe 40 are always minded for the 1st penetrant remover 30 breathed out in the washing port 39. To the penetrant remover reservoir tank 42 side Delivery, Although it is made for the 1st used penetrant remover 30 not to collect in the washing port 39 and the reattachment on the front face of an outside of the needle 37 of a used penetrant remover is prevented, it is not necessary to make it not necessarily operate.

[0020] Thus, if washing of the usual passage is completed, it will move to the step of washing concerning this invention. That is, as first shown in drawing 2 (A), while inserting a needle 37 into the 2nd penetrant remover 44 (it is the same quality of the material as the 1st penetrant remover 30 at this example) filled up in the washing bottle 45, as the drawing 1 solid line shows, between the 2nd and 3rd port b and c is made into a free passage condition for a cross valve 34. By lengthening piston 35a of the measuring syringe 35 in this condition, the 2nd penetrant remover 44 of the specified quantity is inhaled

in the sample solution suction pipe 23.

[0021] And with insertion into the 2nd penetrant remover 44 of a needle 37, the outside front face of a needle 37 will touch the 2nd penetrant remover 44, and the sample solution adhering to the outside front face is washed. In addition, before attracting the 2nd penetrant remover 44 of the above preferably, and a needle 37 more specifically inserts into the 2nd penetrant remover 44, it is making a small amount of air attract by lengthening piston 35a of the measuring syringe 35.

[0022] Subsequently, carry out updrift of the needle 37, that lower limit is made to desert from the oil level of the 2nd penetrant remover 44, and a small amount of air is inhaled by lengthening piston 35a of the measuring syringe 35 further in this condition (refer to this drawing (B)). And you move a needle 37 and make it located in the washing port 39. By pushing piston 35a of the measuring syringe 35 in this condition, the discharge port 39 is made to turn and breathe out the 2nd once attracted penetrant remover 44 from a needle 39 (refer to this drawing (C)), and washing of inside passage is completed. And this 2nd breathed-out penetrant remover 44 is discharged like the 1st penetrant remover 30 of the above outside.

[0023] And if the above-mentioned washing is completed, while suspending a vacuum pump 42, a needle 37 is raised and it is made to desert from the washing port 39. A penetrant remover seems not to carry out the reattachment to the outside front face of a needle 37 in the case of the updrift of a needle 37, since each used penetrant removers 30 and 44 are not storing in the washing port 39 in this example as mentioned above.

[0024] In addition, the washing processing by suction from the above-mentioned needle 37 is repeated two or more times, it may be made to perform it if needed, and, in this case, a cleaning effect goes up more. moreover, the ** to which measurement does not perform washing processing by suction from the needle 37 concerning this invention the degree of capital like this example in using together with the usual washing processing -- every fixed spacing -- or it may be made to carry out in random. Furthermore, although it was made to perform washing concerning this invention in the above-mentioned example after carrying out the usual washing, reverse is sufficient as this sequence.

[0025] Furthermore, the actuation timing of a vacuum pump 42 Before injecting each penetrant removers 30 and 44 from a needle like the example mentioned above, it is not necessary to carry out. Although what is necessary is to complete all the washing processes over a needle 37, to make it operate in front rather than it carries out updrift of the needle 37, and just to be able to discharge the penetrant remover in the washing port 39, preferably Before a used penetrant remover collects in the washing port 39 and a used penetrant remover contacts the lower limit of a needle 27, it is operating a vacuum pump 42.

[0026] Drawing 3 shows the important section of the routing of the 2nd example of this invention. The process which draws in while inserting a needle 37 into the 2nd penetrant remover 44 also by this example, and attracts a small amount of air after carrying out updrift of the needle 37 subsequently is the same as the 1st example of the above. And when the condition in the roppo valve 20 is shown in the sample solution suction-pipe 23 list after attracting the starting air, it is shown in drawing 3 (A). That is, black painting is carried out and the 2nd shown penetrant remover 44 is located to the central part of the sample solution suction pipe 23 to which Port F and the measuring syringe 35 of the roppo valve 20 are connected. In addition, the agreement 47 among drawing is the air which drew in before suction of the 2nd penetrant remover 44.

[0027] In this condition, only the specified quantity pushes piston 35a of the measuring syringe 35. This amount to push is made fewer than the amount subtracted at the last process. Then, as shown in this drawing (B), the 2nd penetrant remover 44 is put back only for the specified quantity. However, with the air which drew in after suction of the 2nd penetrant remover 44 also in this condition although the illustration abbreviation was carried out, from the tip of a needle 37, the 2nd penetrant remover 44 leaks and does not come out.

[0028] And when it push and lengthens and only the count of predetermined operates the above-mentioned piston 35a, drawing 3 (A) and the condition of (B) are repeated. Thereby, the 2nd penetrant remover 44 will carry out both-way migration of the inside of the sample solution suction pipe 23, and a

cleaning effect increases more.
[0029] Then, a needle 37 is moved to the washing port 39 side, and the 2nd penetrant remover 44 inhaled by pushing in piston 35a greatly is discharged. In other configuration lists, since it is the same, the explanation is abbreviated to the 1st example which described the operation effectiveness above.
[0030] In addition, although the penetrant remover which each attracts through a needle 37 explained one kind of thing in each above-mentioned example, this invention is not restricted to this, prepares two or more washing bottles, is filled up with penetrant removers, such as a different class in each washing bottle, for example, alkalinity, acidity, and an organic solvent, and may be made to perform the above-mentioned washing to a desired penetrant remover. By making it the starting configuration, if two or more sorts of penetrant removers are not used like for example, living body samples (blood serum etc.), washing processing can be ensured also to the sample solution which cannot be washed completely.
[0031] And without preparing two or more washing bottles as mentioned above, when two kinds of especially penetrant removers are required, two or more starting sorts and, it is filled up with one penetrant remover in the penetrant remover tank which performs the usual washing, and the same effectiveness can be acquired by being filled up with the penetrant remover of another side in a washing bottle.
[0032] In addition, although each above-mentioned example explained the example which all enforces the washing approach of this invention in addition to the usual washing approach again, it may be made to wash passage etc. only by the washing approach concerning this invention. When starting, a penetrant remover tank can become unnecessary at a cross valve list, direct continuation of the measuring syringe can be carried out to the port of 1 of a roppo valve, and simplification of the equipment for enforcing this invention approach can be attained.
[0033]
[Effect of the Invention] As mentioned above, by the passage washing approach of the automatic sampler concerning this invention, since a needle is inserted into a direct penetrant remover, the outside front face of a needle can contact a penetrant remover, and, thereby, can wash the outside front face. And a penetrant remover is attracted in a sample solution suction pipe through a needle with the condition, and since the attracted penetrant remover is made to flow backwards and it is subsequently made to breathe out outside, washing of the internal passage of a sample solution suction pipe can also be performed. That is, in this invention, since washing can be performed by the same activity as the inhalation process of the sample solution at the time of the usual measurement, the effectiveness that it can be used as it is also does so conventional equipment with special unnecessary equipment and device.

[0034] And by carrying out sequential insertion of the needle, for example into the penetrant remover of a different class, two or more passage can be washed using the penetrant remover of a seed, and passage can be washed more certainly. Washing processing can be ensured even if it is after measurement of the sample solution which cannot wash completely, if it follows, for example, two or more sorts of penetrant removers, such as a blood serum and urine, are not used. Consequently, since it does not stick to the Teflon (trademark) in which the protein and the lipid in the blood serum to apply, urine, etc. form each passage, such as a sample solution suction pipe, the volume in passage (cross section) can perform exact measurement, without intermingling the sample solution of the measuring object, and the sample solution performed last time, while always being kept constant. Furthermore, it can measure continuously also to the required sample solution of a different penetrant remover, and working efficiency improves.

[Translation done.]

What is claimed is:

1. An automatic sampler for injecting a sample into a sample introducing portion in communication to a column of a liquid chromatography, comprising:

a) a needle for sucking the sample from a sample liquid bath and injecting the sample into the sample introducing portion;

b) a first rinsing section for rinsing said needle by soaking said needle in a first rinsing liquid, in which the first rinsing liquid is not exchanged during the rinsing operation; and

c) a second rinsing section for rinsing said needle by soaking said needle in a second rinsing liquid, in which the second rinsing liquid is exchanged during the rinsing operation,

wherein said needle is rinsed by at least one rinsing section selected from said first rinsing section and said second rinsing section.

2. The automatic sampler according to claim 1, further comprising:

a switching section for selecting the second rinsing liquid for use with said second rinsing section from among a plurality of rinsing liquids.

3. A method for rinsing a needle of an automatic sampler,

said method comprising:

rinsing the needle by at least one operation selected from a) rinsing the needle by soaking the needle in a first rinsing liquid, in which the first rinsing liquid is not exchanged during the rinsing operation, and b) rinsing the needle by soaking the needle in a second rinsing liquid, in which the second rinsing liquid is exchanged during the rinsing operation.

4. The method for rinsing a needle of an automatic sampler according to claim 3, further comprising:

selecting the second rinsing liquid from among a plurality of rinsing liquids.